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Baseline survey of marine sediments collected from the State of Kuwait: PAHs, PCBs, brominated flame retardants and metal contamination

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ABSTRACT

A geographically extensive baseline survey of sediment contamination was undertaken at twenty nine locations around Kuwait. Samples were assessed in relation to a wide range of industrial pollutants, including metals, PAHs, PCBs, PBDEs and HBCDs. The data generated indicated that levels of pollutants were generally low and below commonly applied sediment quality guidelines (SQGs). However, naturally high background concentrations of certain metals present in sediment from the region may prohibit the direct assessment against some of the routinely applied SQGs. Hot spots of contamination were identified for PAHs, PCBs and PBDEs, that were mainly associated with the Shuaiba Industrial Area, located south of the city, and known to contain a diverse mix of both light and heavy industry.

1. Introduction

The State of Kuwait has witnessed major economic, social and industrial development following the discovery and exploitation of its vast oil reserves (Al-Abdulghani et al., 2013). Similar to other countries, which comprise the Gulf Co-operative Council (GCC), the rapid expansion of Kuwait's industrial sector has mainly occurred around its coasts (Al-Rifaie et al., 2007; Al-Abdulghani et al., 2013). As a consequence a variety of contaminants have been discharged directly into the marine environment, including petroleum hydrocarbons, trace metals, nutrients (from raw domestic sewage), and contaminated brine from desalination plants, which are essential for freshwater production in the region (Readman et al., 1992; Al-Ghadban et al., 2002; Al-Sarawi et al., this issue). Analysis of sediment and biota have shown the marine environment around Kuwait to be contaminated with a range of aliphatic and polycyclic aromatic hydrocarbons (PAHs) and organochlorine contaminants (Beg et al., 2009; de Mora et al., 2010; Al-Sarawi et al., this issue). Power generating industries and desalination plants are also known to be point sources of contamination and elevated levels of heavy metals, which in some instances exceeded human consumption safety limits, have been observed in clams (*Amiantis umbonella*) collected from Kuwait Bay (Tarique et al., 2012, 2013). A large number of industrial outfalls, storm water culverts and earth channels are situated along the coastline of Kuwait and discharge directly into the sea. It is known that these release raw sewage and untreated industrial water to the marine environment (Ghannoum et al., 1991; Al-Ghadban et al., 2002; Bu-Olayan and Thomas, 2014; Lyons et al., this issue). Domestic sewage in Kuwait has a high organic content and is often septic because of low flows, long retention times, high ambient temperatures and concomitant anaerobicity (Al-Ghadban et al., 2002).

Past events, such as the 1991 Gulf War, have further contributed to environmental pressures associated with rapid industrialization. During this period it is estimated that 9–10.8 million barrels of oil were released into the coastal waters of Kuwait from sabotaged tankers and pipelines at the Al-Ahmadi terminal (Al-Abdali et al., 1996; Readman et al., 1996). As a consequence the environment was exposed to an array of contaminants, which included petroleum hydrocarbons from burning oil wells and polychlorinated biphenyls (PCBs) and heavy metals from damaged industrial facilities (Massoud et al., 1998; Al-Sarawi et al., 2002). These impacts are exacerbated by other sources of marine pollution that include atmospheric fallout from dust storms and particulate matter transported from the Shatt Al-Arab river (Al-Ghadban et al., 2002; Al-Ghadban and El-Sammak, 2005). It is also known that natural oil

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seepage occurs at a number of sub-sea locations and these are also thought to be important point sources of contamination at various locations around the coast (Al-Ghadban et al., 2002).

Here we describe the results from a survey which collected sediment from twenty nine locations situated within Kuwait Bay (including Sulaibikhat Bay) and along the Gulf coastline towards the Shuaiba Industrial Area (SIA) to the south of the city (Fig. 1). Sediments were analysed for PAHs, metals and an array of organohalogen compounds.

2. Material and Methods

2.1. Sampling and characterisation of marine sediment from Kuwait

Samples were collected during 2013/2014 using a hand-held van Veen grab deployed from research vessels provided by the Kuwait Environment Public Authority (KEPA), Kuwait Institute of Scientific Research (KISR) and Public Authority for Agriculture and Fish Resources (PAAFR). The top layer of each grab sample was collected using a stainless steel scoop and immediately transferred to a *n*-hexane rinsed 500 ml glass jar. Samples were kept on ice before transferring to <−20 °C for storage prior to analysis. Particle size analysis (PSA) was completed on each sample. Laser diffraction analysis of the <1 mm fraction of each sample was undertaken, with the remaining sediment wet split at 63 μm. The >63 μm fraction was dry sieved at 0.5 Phi intervals down to 4 Phi (63 μm). The <63 μm fraction was freeze-dried and weighed. The sieve and laser diffraction data were merged to form a complete particle distribution for each sample. Quality assurance tests include comparing weights of samples before sieving, during sieving and after sieving. Totals are checked and any results with any anomalies were re-sieved. Certified reference materials (CRMs) were analysed regularly with the laser-sizer, as well as an in-house reference material (IHRM) which was analysed at the start of every day that analysis was completed. PSA ring tests are completed as part of the National Marine Biological Analytical Quality Control (NMBAQC) scheme. Total organic carbon (TOC) was completed on both the <63 μm fraction (prepared as described within PSA methodology) and <2 mm fraction. For the <2 mm fraction, sediment samples were air-dried after removal of >2 mm fraction. The <2 mm sediment was ground and both <63 μm fraction and <2 mm fractions analysed. Inorganic carbon was removed using a sulphurous acid digest. TOC was measured using a Carlo Erba EA1108 Elemental analyser. Quality control was carried out with 3 repeats for 1 in 10 samples (with rsd% of <10%), additionally a CRM was included for 1 in 10 samples and 3 replicates of IHRM were completed for each batch. Limits of detection were <0.02% for organic and

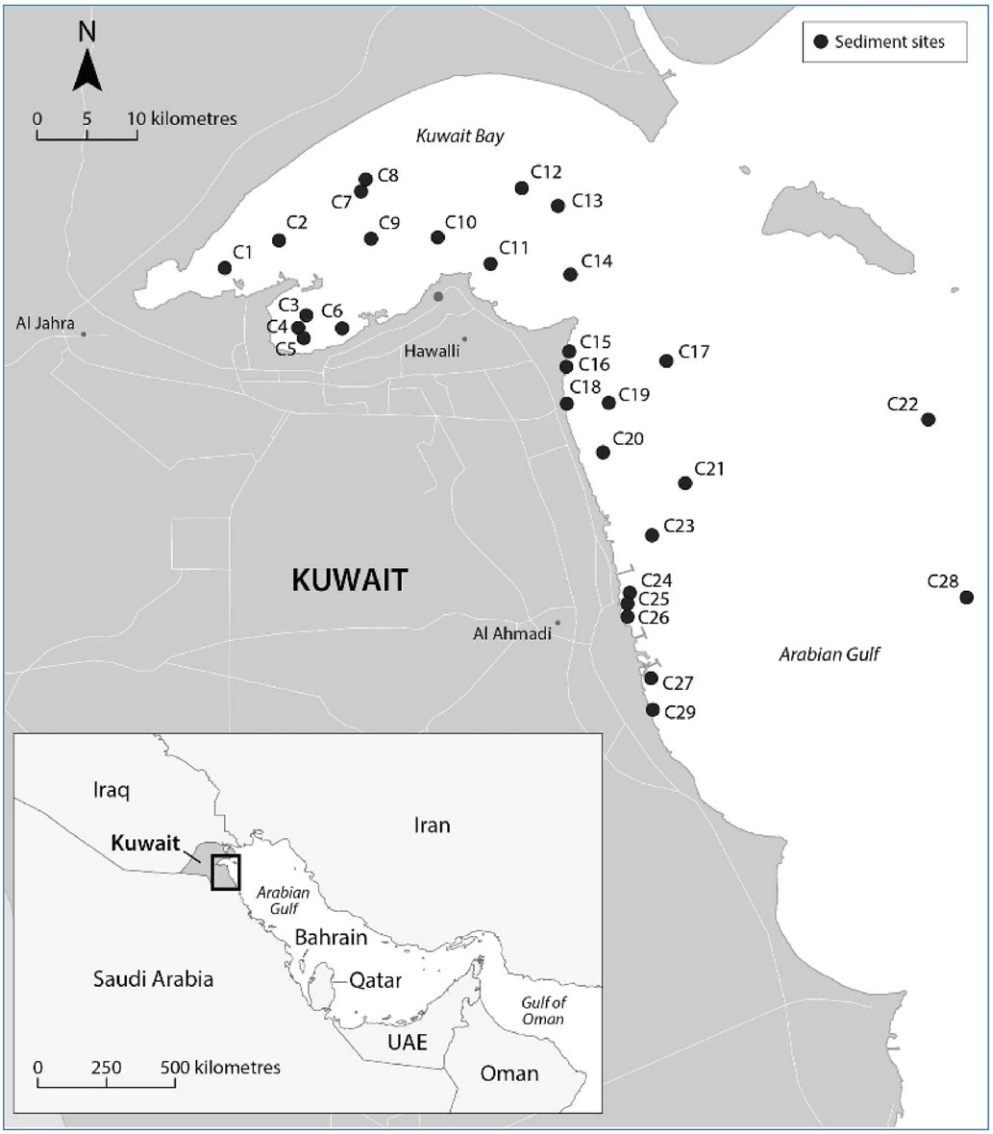

Fig. 1. Sediment sampling locations.

<0.002% for nitrogen, and measurement limits were calculated as 10 times detection limit. Sediment characteristics, including PSA and TOC analysis are displayed in Table 1.

2.2. Sediment metal analysis

The fine sediment fraction sample (<63 μm) was digested in a mixture of hydrofluoric, hydrochloric and nitric acids using an enclosed vessel microwave (MarsXpress Microwave Reaction System, CEM Ltd, Buckingham, UK). Typically, approximately 0.2 g of sample was weighed out and pre-digested overnight in a mixture of nitric acid, hydrochloric acid and hydrofluoric acid (HF) (Aristar grade 69%, VWR, Leicestershire, UK). The excess of HF was complexed with the addition of a saturated boric acid solution. The digest was then further diluted prior to analysis by inductively coupled plasma-mass spectrometry (ICP-MS, Agilent 7500ce, Agilent Technologies, Waldbronn, Germany) and by inductively coupled plasma-optical emission spectroscopy (ICP-OES, Thermo iCAP 6500 Duo, Thermo Scientific, Hemel Hempstead, UK). Quantification of trace elements was performed by external calibration using 8 calibration levels (0–500 ng ml^{-1}), using indium as internal standard.

2.3. Sediment PAH analysis

Each homogenised wet sediment sample was spiked with an analytical surrogate consisting of a suite of deuterated PAHs (naphthalene-d_8, acenaphthylene-d_8, anthracene-d_{10}, dibenzothiophene-d_8, pyrene-d_{10}, benzo[a]anthracene-d_{12}, benzo[a]pyrene-d_{12} and dibenz[a,h]anthracene-d_{14}) and extracted by alkaline saponification in methanolic potassium hydroxide followed by liquid/liquid solvent extraction using glass-distilled grade pentane and drying of the extracts with sodium sulphate. The total hydrocarbon concentration in the extracts was determined by means of ultra-violet fluorescence spectrometry as a screen of the level of contamination. Sub-samples of wet sediment were dried to measure total solids percentage, which was used to enable the calculation of PAH concentrations on a dry weight (dw) basis.

An aliquot of each sediment extract was concentrated and passed through a short alumina chromatography column and eluted in dichloromethane:pentane [1:1] in order to clean-up the extract. The cleaned up extract was concentrated to 1 ml and analysed for a suite of parent and alkylated PAHs by gas chromatography-mass spectrometry in electron impact ionization mode (GC-MS) using a 6890 GC coupled to a 5975 MSD (Agilent Technologies, Walbron, Germany) in synchronous multiple ion detection/full scan, monitoring the molecular ions of each compound or group (the latter in the case of the alkylated PAH) determined. Aliquots of the extracts (2 μL) were analysed using a DB-5.625 (30 m × 0.25 mm ×0.25 μm) cross linked fused silica capillary column coated with 95% dimethyl 5% diphenyl polysiloxane (J&W Scientific, Folsom, CA, USA). The carrier gas was helium at a constant flow of 1 ml/min. Injection was in pulsed splitless mode with an injector temperature of 300 °C. The injection was made with the column at 60 °C and following injection the oven temperature was held at 60 °C for 2 min and subsequently raised to 310 °C at a rate of 5 °C/min where it was held for 10 min giving a standard run time of 62 min. The GC was directly coupled to the MS detector via a transfer line heated to 310 °C. The mass spectrometer was operated in SIM/full scan mode and scans were from 35 to 325 Da. Quantification for PAHs was performed using surrogate standards and 5 calibration levels (range 25–500 ng ml^{-1}).

2.4. Sediment organohalogen analysis

Sediment samples were air dried and sieved (<2 mm) in a controlled environment. 10 g of dried sediment were mixed with sodium sulphate, transferred to a glass Soxhlet thimble and topped with 1 cm of sodium sulphate. The samples were subjected to Soxhlet extraction using acetone: *n*-hexane 1:1 (*v*:*v*) for approximately 6 h. Prior to extraction, the samples for BDE209 analysis were spiked with $^{13}C_{12}$-BDE209. Sulphur residues were removed at this stage with copper filings.

Sediment extracts for hexabromocyclododecane (HBCDD) analysis were spiked with an analytical surrogate (consisting of d_{18}-α-, d_{18}-β-, d_{18}-γ-HBCDD and $^{13}C_{12}$-tetrabromobisphenol-A, TBBPA) and cleaned

Table 1
Sediment characteristics, PSA, TOC and EUNIS sediment classification. ns: no sample.

Site name	Region	EUNIS sediment classification	<63 μm: Organic carbon (% m/m)	<63 μm: Nitrogen (% m/m)	<2 mm: Organic carbon (% m/m)	<2 mm: Nitrogen (% m/m)	<2 mm: Carbon:nitrogen ratio
C1	Kuwait Bay	Mud and sandy mud	1.75	0.23	1.47	0.23	6.39
C2	Kuwait Bay	Mixed sediments	1.02	0.15	0.96	0.15	6.40
C3	Sulaibikhat Bay	Mixed sediments	0.89	0.12	0.69	0.13	5.30
C4	Sulaibikhat Bay	Mud and sandy mud	1.08	0.10	0.66	0.12	5.50
C5	Sulaibikhat Bay	Mud and sandy mud	0.81	0.11	0.71	0.12	5.92
C6	Sulaibikhat Bay	Sand and muddy sand	1.50	0.22	0.62	0.09	6.89
C7	Kuwait Bay	Mud and sandy mud	0.54	0.09	0.58	0.11	5.27
C8	Kuwait Bay	Mud and sandy mud	0.74	0.14	0.73	0.16	4.56
C9	Kuwait Bay	Mud and sandy mud	1.07	0.14	0.97	0.16	6.01
C10	Kuwait Bay	Mixed sediments	1.07	0.12	0.69	0.12	5.75
C11	Kuwait Bay	Mixed sediments	1.00	0.12	0.68	0.12	5.67
C12	Kuwait Bay	Mud and sandy mud	1.12	0.10	0.67	0.11	6.09
C13	Kuwait Bay	Mud and sandy mud	0.70	0.14	0.57	0.12	4.75
C14	Kuwait Bay	Mud and sandy mud	0.81	0.10	0.64	0.11	5.82
C15	Gulf coast	Sand and muddy sand	ns	ns	0.20	0.05	4.00
C16	Gulf coast	Sand and muddy sand	ns	ns	0.22	0.05	4.40
C17	Gulf coast	Mud and sandy mud	0.67	0.15	0.61	0.14	4.36
C18	Gulf coast	Mixed sediments	1.35	0.15	0.66	0.09	7.33
C19	Gulf coast	Mixed sediments	0.84	0.11	0.63	0.11	5.73
C20	Gulf coast	Mixed sediments	0.95	0.14	0.63	0.09	7.00
C21	Gulf coast	Mud and sandy mud	0.64	0.13	0.55	0.13	4.23
C22	Gulf coast	Mud and sandy mud	0.74	0.15	0.78	0.15	5.20
C23	Gulf coast	Mud and sandy mud	0.89	0.14	0.80	0.14	5.71
C24	Gulf coast	Mixed sediments	1.65	0.23	1.47	0.13	11.31
C25	Gulf coast	Coarse sediment	ns	ns	0.20	0.08	2.50
C26	Gulf coast	Mud and sandy mud	1.57	0.19	1.25	0.14	8.93
C27	Gulf coast	Coarse sediment	ns	ns	1.73	0.11	15.73
C28	Gulf coast	Mud and sandy mud	0.96	0.17	0.92	0.18	5.11
C29	Gulf coast	Coarse sediment	ns	ns	2.85	0.12	23.75

up by high resolution gel permeation chromatography (HRGPC), using a series 1100 HPLC system (Agilent Technologies, Waldbronn, Germany), followed by acid silica chromatography. The HRGPC columns used were two Envirogel TM columns (Waters Corporation, Milford, MA, USA; 150 mm × 19 mm i.d. and 300 mm × 19 mm i.d.), connected in series and protected by an Envirogel TM guard column (Law et al., 2006). For the acid silica chromatography step, concentrated HRGPC fractions were eluted through 8 g of 45% acid silica with 25 ml of 1:1 dichloromethane:hexane (Harrad et al., 2009). Three diastereoisomers, α-, β- and γ-HBCDD, and TBBP-A were determined using ultra performance liquid chromatography (UPLC; Acquity, Waters) tandem mass spectrometry (TQ MS/MS; Xevo TQ MS, Waters). Compound separation was achieved on a BEH C_{18} UPLC column (1.7 μm, 2.1 × 50 mm; Waters) using a gradient programme from 30%:70% to 0.1%:99.9% 2 mM ammonium acetate buffered water:acetonitrile. Quantitation for HBCD and TBBP-A was performed using isotope dilution and 8 calibration levels (range 0.5–200 ng ml^{-1}).

An aliquot of the sediment extracts was cleaned up and fractionated using alumina (5% deactivated) and silica (3% deactivated) columns, respectively. The silica column fractionation results in two fractions, the first fraction containing polychlorinated biphenyls (PCBs) and BDE209, the second fraction containing other polybrominated diphenylethers (PBDEs). The final GC-ready fractions were spiked with PCB53 (for PCBs analysis) and PCB200 (for PBDE analysis) and made up to a final volume of 1 ml.

PCB concentrations in sediment samples were determined with an Agilent 6890 GC with μECD (Agilent Technologies, Waldbronn, Germany). The separation of analytes was performed on a 50.0 m × 200 μm, 0.33-μm-film-thickness DB-5 capillary column (J&W). The carrier and ECD make-up gas were hydrogen (32.2 psi constant pressure, initial velocity 50 cm/s) and argon/methane (95:5), respectively. The initial oven temperature was 90 °C, held for 2.00 min, then increased to 165 °C at 15 °C/min, to 285 °C at 2 °C/min, and finally held for 23 min. The injector temperature and detector temperature was 270 °C and 300 °C, respectively. A 1 μL extract was injected in splitless mode with a purge time of 2 min. The PCB standard solutions contained the following 27 compounds in iso-octane: Hexachlorobenzene; *p,p′*-DDE; CB18; CB28; CB31; CB44; CB47; CB49; CB52; CB66; CB101; CB105; CB110; CB118; CB128; CB138; CB141; CB149; CB151; CB153; CB156; CB158; CB170; CB180; CB183; CB187; and CB194, together with the internal standard CB53. Quantitation was performed using internal standards and 7 calibration levels (range 0.5–100 ng ml^{-1}).

PBDE congeners were determined by gas chromatography-mass spectrometry in electron capture negative ionization (GC-MS-ECNI) (de Boer et al., 2001) with an Agilent 6890 GC with 5973 MS (Agilent Technologies, Waldbronn, Germany) in negative chemical ionisation (NCI) mode. The separation of analytes was performed on a 50.0 m × 250 μm, 0.25-μm-film-thickness DB-5 capillary column (Agilent Technologies J&W columns). The carrier gas was helium (30 psi constant pressure, average velocity 40 cm/s) and the reagent gas was methane (40 psi). The initial oven temperature was 90 °C, held for 2.00 min, then increased to 200 °C at 30 °C/min, to 295 °C at 2.5 °C/min, and finally held for 31.3 min. The injector temperature and detector temperature was 270 °C and 200 °C, respectively. A 2 μL extract was injected in splitless mode with a purge time of 2 min. Quantitation for PBDEs was performed using internal standards and 8 calibration levels (range 0.1–50 ng ml^{-1}). The PBDE standard solutions contained the following 11 compounds in iso-octane: BDE17; BDE28; BDE47; BDE66; BDE85; BDE99; BDE100; BDE138; BDE153; BDE154; and BDE183; together with the internal standard CB200. BDE209 concentrations were determined with an Agilent 6890 GC with 5973 MS (Agilent Technologies, Waldbronn, Germany) in NCI mode using $^{13}C_{12}$-BDE209 as internal standard. The separation of analytes was performed on a 15.0 m × 250 μm, 0.1-μm-film-thickness DB-1 capillary column (J&W). The carrier gas was helium (1.3 ml/min constant flow, average velocity 59 cm/s) and the reagent gas was methane (40 psi). The initial oven temperature was 90 °C, held for 1.00 min, then increased to 200 °C at 25 °C/min, to 295 °C at 10 °C/min, and finally held for 20 min. The injector temperature and detector temperature was 250 °C and 200 °C, respectively. A 2 μL extract was injected in pulsed splitless mode with a 20 psi pulse until 1 min and a purge time of 2 min. Quantitation of BDE209 was performed using an internal standard and 7 calibration levels (range 0.5–500 ng ml^{-1}).

2.5. Quality assurance and quality control

The laboratory biannually participates in the Quasimeme (Quality Assurance of Information for Marine Environmental Monitoring in Europe) proficiency testing scheme as external quality assurance. For internal quality assurance, all analyses were carried out under full analytical quality control procedures that included the analysis of certified reference material(s) and a procedural blank sample with every batch samples analysed so that the day-to-day performance of the methods could be assessed. If levels of target analytes in the samples were outside of the range of the instrument calibration, extracts were diluted to be within range and re-analysed. Reference materials used were NIST-1944 (New Jersey Harbour sediment; National Institute of Standards and Technology, Gaithersburg, USA), PACS-2 (marine sediment, National Research Council Canada, Ontario, Canada) and TH2 (harbour sediment, Environment Canada, National Water Research Institute, Ontario, Canada). The results obtained for the reference materials were plotted as Shewhart quality control charts for each compound or trace element determined. The charts had previously been created by the repeated analysis of the above certified reference materials in the Cefas Lowestoft Laboratory using the North West Analytical Quality Analyst software™ (Northwest Analytical Inc., USA). Warning and control limits had been defined for the charts as 2σ and 3σ–2 and 3 times the standard deviation from the mean for each compound or trace element, respectively. The results obtained for all samples analysed were accepted as valid as the results for the certified reference materials were within the limits set by the control charts.

3. Results and discussion

3.1. Sediment metal contamination

Results from the sediment analysis for trace metals are presented in Table 2 and Supplementary data file 1. The concentration of trace metals Cr (108.2–429.0 μg g^{-1} dw), Ni (86.2–169.3 μg g^{-1} dw), Cu (21.7–53.4 μg g^{-1} dw), Zn (55.3–140.7 μg g^{-1} dw), As (3.1–7.4 μg g^{-1} dw), Cd (<0.178–0.5 μg g^{-1} dw), Pb (9.7–32.2 μg g^{-1} dw) and Hg (<0.097–<0.236 μg g^{-1} dw) were assessed against the Background Assessment Concentrations (BACs) and Effects Range Low/Effects Range Median (ERL and ERM) concentrations. BACs were developed by the Oslo and Paris Commission (OSPAR) for testing whether concentrations are near background levels (OSPAR, 2008) and ERL/ERM concentrations, which were developed for the US EPA, are founded on a large database of sediment toxicity and benthic community information (Long et al., 1995; Long and MacDonald, 1998). The ERL/ERM methodology derives Sediment Quality Guidelines (SQGs) representing, respectively, the 10th and 50th percentiles of the effects dataset. This approach is a reasonably conservative one, and has been partially validated using North American field data. Concentrations below the ERL rarely cause adverse effects in benthic marine organisms. All stations depicted levels of Ni above the ERM concentrations. This observation indicates either the mineralogical background is naturally high in Ni or the sampling area is highly enriched with Ni due to surrounding industrial activities over the years. Station C16 showed Cr concentration above ERM level, this station is situated north of a known sewage outfall. The remaining stations exhibited levels of Cr above ERL concentration but remained below ERM level. Most of the stations that showed levels of Cu above

Table 2
Levels of metal contamination (μg g^{-1} dw) in marine sediments collected from Kuwait. Assessments conducted alongside OSPAR BAC (OSPAR, 2008), ERLs/ERM (Long and MacDonald, 1998) and ISQG/PEL (CCME, 1999) criteria.

Site name	Cr	Ni	Cu	Zn	As	Cd	Pb	Hg
C1	212.6	169.1	49.5	123.1	6.9	0.5	14.3	<0.109
C2	148.6	169.3	44.0	111.5	4.9	0.3	13.8	<0.106
C3	241.3	142.2	34.6	100.0	4.9	0.3	10.8	<0.113
C4	267.2	126.2	29.5	91.6	3.6	0.2	11.8	<0.113
C5	176.7	122.3	30.4	90.8	4.0	0.3	11.3	<0.104
C6	162.1	138.2	41.7	108.9	4.5	0.3	13.8	<0.113
C7	181.4	166.5	31.9	87.3	5.4	0.3	10.5	<0.102
C8	157.9	142.1	27.3	67.5	4.2	0.3	13.8	<0.222
C9	154.6	157.5	37.9	103.9	4.0	0.3	12.6	<0.108
C10	182.4	159.0	34.0	99.7	4.9	0.2	12.7	<0.12
C11	202.5	147.4	29.7	92.4	5.3	0.3	12.3	<0.102
C12	152.6	145.3	26.8	81.1	4.6	0.3	10.3	<0.108
C13	144.0	127.8	22.6	66.8	4.2	0.2	12.6	<0.204
C14	159.8	148.1	23.9	69.4	5.0	0.3	9.7	<0.097
C15	183.6	144.7	53.4	140.7	7.2	<0.391	15.9	<0.235
C16	429.0	121.8	34.7	98.4	5.4	0.3	15.3	<0.113
C17	140.6	135.8	25.4	67.6	4.2	0.2	12.9	<0.213
C18	171.6	140.1	33.8	90.8	4.2	0.3	13.0	<0.114
C19	154.2	147.4	28.0	85.7	3.8	0.2	11.0	<0.112
C20	155.2	142.3	49.6	108.9	3.4	0.2	14.9	<0.113
C21	145.9	119.7	21.7	55.3	5.3	0.2	11.8	<0.214
C22	125.3	129.5	24.6	66.5	3.1	<0.192	13.7	<0.23
C23	148.8	161.6	30.4	85.1	4.9	0.3	11.8	<0.112
C24	144.7	122.4	39.6	90.3	3.5	<0.178	19.1	<0.213
C25	135.1	120.6	38.7	82.4	5.2	0.2	33.2	<0.236
C26	108.2	86.2	41.1	80.9	5.2	0.2	20.7	<0.228
C27	135.1	124.6	51.9	94.2	7.4	0.2	29.8	<0.235
C28	143.7	149.9	29.2	74.7	3.7	0.3	12.6	<0.229
C29	228.0	109.2	28.3	78.7	4.4	<0.183	17.4	<0.219
Assessment criteria								
OSPAR BAC	81	36	27	122	25	0.31	38	0.07
ERL	81	20.9	34	150	8.2	1.2	46.7	0.15
ERM	370	51.6	270	410	70	9.6	218	0.71
ISQG	52.3	–	18.7	124	7.24	0.7	30.2	0.13
PEL	160	–	108	271	41.6	4.2	112	0.7

ERL (but below ERM concentration) are located by the shoreline. There appears to be a decreasing gradient in concentrations as the stations are further offshore, indicating a possible dilution effect from industrial activities. The majority of the stations recorded levels of Zn and Cd below the OSPAR BACs, with a few stations depicting levels below ERL concentrations. Interestingly, levels of As and Pb were below OSPAR BACs for all stations. Concentrations of Hg were below the method limit of detection for all stations, indicating levels of Hg either below ERL or below OSPAR BACs. In general, no difference was observed when concentrations recorded from stations located near industrial areas (e.g C1, C24–C27) were compared against levels of metals from residential areas (e.g C15–C18).

Previous studies have used sediment core data to estimate the natural background levels of metals present in different regions of the Gulf's marine environment (Al — Abdali et al., 1996). Using this approach, background levels were proposed for: Zn 30–60, Pb 15–30, Cd, 1.2–2.0, Ni 70–80, Mn 300–600, Fe 10,000–20,000, V 20–30, and Cu 15–30 μg g^{-1} dw (Al-Abdali et al., 1996). The levels of sediment contamination by metals in this present study are similar to those previously reported for Kuwait's marine environment. For example, the concentrations of metal reported by Metwally et al. (1997) included hot spots of contamination around known anthropogenic inputs (Ni range: 12.3–235.6; Pb range 71.55–261.4 and V range: 24.8–179.41 μg g^{-1} dw). Al-Sarawi et al. (2002) and Alshemmari et al. (2010) carried a similar assessment of trace metal pollution in bottom sediments collected from of Sulaibikhat Bay, which corresponds to sites C3–C6 in the present study. Broadly similar concentrations of metals were again reported and while the authors noted that concentrations of some metals were clearly within the natural background levels, as previously reported by Al-Abdali et al. (1996), the values of others exceeded a number of published SQGs (Alshemmari et al., 2010).

The data present in this current study along with that of Alshemmari et al. (2010) highlights the caution that must be applied when applying commonly used SQGs, such as ERLs and ERMs proposed by NOAA (Long and MacDonald, 1998) or Interim Sediment Quality Guideline (ISQG) and Probably Effect Levels (PEL) proposed by CCME (1999). The data presented clearly shows that for selected metals the background concentrations for the region as proposed by Al-Abdali et al. (1996), actually exceed the established ERL/ERM or ISQG/PEL criteria. For example, in the current study the values of Ni recorded across the whole of Kuwait range from 86.2–169.34 μg g^{-1}, which exceeds the ERL (20.9 μg g^{-1}) and ERM (51.6 μg g^{-1}) SQGs. However, the proposed background concentration for Ni for the Gulf region has been set at 70–80 μg g^{-1}, which clearly is above both the ERL and ERM threshold. A similar situation also occurs for Cu and Cd, where proposed background concentrations exceed the ERL and/or ISGL. Therefore, until a fully validated series of region specific guidelines are developed the current ERLs/ERMs or ISQG/PEL should purely be used to indicate levels below which biological effects are not thought to occur and any exceedance need to be investigated further taking into account natural background levels of contamination and the sensitivity of resident marine species.

3.2. Sediment total hydrocarbon content (THC) and PAH concentrations

Results from the sediment analysis for THC and PAHs are presented in Table 3 and Supplementary data file 2 and Supplementary data file 3. The concentration of THC recorded in the present study (range: 4.2–744 μg g^{-1} dw) match those previously reported for the region (Fowler et al., 1993; Readman et al., 1996; Metwally et al., 1997; de

Table 3
Total hydrocarbon content (THC), Low Molecular Weight (LMW: naphthalene, methyl naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene) and High Molecular Weight (HMW: fluoranthene, pyrene, benz[a]anthracene, chrysene, benzo[a]pyrene, dibenz[a,h]anthracene) PAH and ∑PAH concentrations from marine sediments around Kuwait. ERL and ERM taken from Gorham-Test et al. (1999).

Site	THC μg g^{-1} dw	∑LMW ng g^{-1} dw	∑HMW ng g^{-1} dw	∑30PAH ng g^{-1} dw	% Oil	% Combustion
C1	39.0	13.2	15	190.1	72	28
C2	20.0	8.5	13	102.9	57	43
C3	18.0	5.6	6	29.2	39	61
C4	23.0	10.8	14	58.7	44	56
C5	32.0	13.6	17	101.0	60	40
C6	13.0	3.4	11	50.9	56	44
C7	4.2	6.3	3	45.9	74	26
C8	13.0	8.7	5	74.7	71	29
C9	19.0	7.4	10	85.0	60	40
C10	27.0	8.3	11	89.6	58	42
C11	30.0	8.7	9	91.7	63	38
C12	15.0	4.5	6	53.7	58	42
C13	9.0	4.2	4	66.1	57	43
C14	12.0	7.8	7	75.4	57	43
C15	9.1	6.5	1	18.1	89	11
C16	17.0	7.8	2	36.5	79	21
C17	10.0	8.3	5	74.0	65	35
C18	32.0	6.9	5	45.9	65	35
C19	15.0	9.0	7	73.9	63	38
C20	12.0	3.4	3	34.3	56	44
C21	7.0	4.2	3	40.7	59	41
C22	19.0	8.4	7	78.3	60	40
C23	41.0	8.3	18	115.7	45	55
C24	105.0	7.7	43	192.4	33	67
C25	17.0	3.7	54	140.8	28	72
C26	744.0	36.2	277	1286.0	35	65
C27	32.0	21.2	22	122.0	45	55
C28	29.0	10.2	6	97.7	67	33
C29	4.7	1.9	2	12.9	40	60
Assessment criteria						
ERL	–	552	1700	–	–	–
ERM	–	3160	9600	–	–	–

Mora et al., 2010). It is important to note that this must be viewed with a degree of caution, as while comparisons of THC/Total Petroleum Hydrocarbon (TPH) are often made between studies the values are often derived using different extraction and analytical techniques. Previous work conducted to map the extent of oil contamination several years after the 1991 Gulf War reported TPH concentrations ranging from 40–240 μg g^{-1} dw (Fowler et al., 1993; Readman et al., 1996). Similar concentrations were reported by Metwally et al. (1997) who in a wider spatial survey reported TPH contaminations ranging between 7.43 μg g^{-1} in reference sediments to 458.61 μg g^{-1} at sites close to known sources of pollution around the Shuaiba Industrial Area (SIA). Beg et al., 2003 also reported high concentrations of TPH at sites close to the SIA (6.7–2066.9 μg g^{-1}). The general findings of Metwally et al. (1997) and Beg et al. (2003) match the findings of the current survey, which clearly demonstrates elevated THC concentrations at the sites, C24 and C26, located close to the SIA. If we exclude these two locations in close proximity to the SIA, the concentration of THC at the remaining sites in the present study range between 4.2 and 41 μg g^{-1}, which closely match the findings of Massoud et al. (1998) who undertook an extensive survey of the whole Arabian Gulf and categorised the majority of Kuwait's offshore sediments to be only slightly contaminated with TPHs (15–50 μg g^{-1}).

Summed PAH concentrations (total of 30 compounds including parent and alkylated PAHs) were compared at all the sites (Table 3; Supplementary data file 2; Supplementary data file 3). The highest ∑30PAH concentration was observed in a muddy sediment at site C26 with 1290 ng g^{-1} dw. This was found to be made up from approximately 70% PAHs derived from combustible sources and was located close to the SIA, where there is a presence of cement factories and oil related industries. The next highest value observed in this survey was found at sample site C24 (also close to the SIA), characterised as a muddy shell sample with ∑30PAH concentrations of 192 ng g^{-1} dw. Similar values were found at sample site C1, characterised as a watery mud sediment sample with ∑30PAH concentration of 190 ng g^{-1} dw derived from approximately 70% oil originated source, whilst C2, C5 C23, C25 C27 and C28 were found to be between 98 and 141 ng g^{-1} dw. All other sample sites had ∑30PAH below 100 ng g^{-1} dw, with concentrations of 13 ng g^{-1} dw in sandy shelly sediment at C29, south of SIA and concentrations of 18 ng g^{-1} dw in sandy sediment at C15 which is in a predominantly residential area. However, despite having low concentrations the source of PAHs found at sites C1, C7, C8 and C16 also were shown to be greater that 70% oil derived and approximately 90% oil derived at C15. Interpretation of spectral data shows signs of weathered and degraded oil that has originated from different industrial sources of oil as well as some refined oil, possibly originating from the first Gulf War spill in the 1990s. By looking at the carbon preference index (CPI) we can assess whether the origin of n-alkanes is petrogenic or natural based produced from marine algae or a mixture of the two sources. Our observations indicate that the highest CPI is 1.35 meaning there is a greater proportion of natural n-alkanes than petrogenic. This is characterised by higher levels of >C20 n-alkanes which indicate natural inputs from off the land. Whilst there appears to be little vegetation along the margin of Kuwait the natural source is likely to have originated from salt grasses and marshes in Iraq via the Shatt al Arab waterway rather than from the marine algae which would be indicated by higher ratio in the C17–C19 n-alkane profiles. Two previous studies also documented individual and ∑PAH concentrations in marine sediments collected in Kuwaiti waters (Beg et al., 2003; de Mora et al., 2010). While the constituent ∑PAHs differed slightly between studies the general levels of contamination were considered similar with 5.65–1333.6 ng g^{-1} dw and 12 to 1670 ng g^{-1} dw reported by Beg et al. (2003) and de Mora et al. (2010) respectively. Again, and similar to the data presented in the current study, the most contaminated sites were those close to the SIA with individual PAHs such as phenanthrene (maximum value recorded: 165.5 ng g^{-1} dw), fluoranthene (maximum value recorded: 292.57 ng g^{-1} dw) and benzo[a]pyrene (maximum value recorded: 94.75 ng g^{-1} dw), reported by Beg et al. (2003). Such values are still relatively low when compared to other industrialised locations around the world where ∑PAH concentrations can exceed 40,000 ng g^{-1} dw (Woodhead et al., 1999; Nicolaus et al., 2015).

As for the assessment of metal contamination we applied the ERL/ERM methodology, as proposed by NOAA, to the current PAH sediment data (Long et al., 1995; Long and MacDonald, 1998). In a regulatory context, where SQGs are to be used as informal (non-regulatory) benchmarks to aid in the interpretation of sediment chemistry (Long et al., 1995), this becomes complicated when a large number of individual PAH compounds are determined, as is usually the case. This has led to separate ERL/ERM derived SQGs being set for "Low Molecular Weight (LMW) PAHs" and "High Molecular Weight (HMW) PAHs" (Gorham-Test et al., 1999). In this context, LMW PAH includes the 2- and 3-ring PAH compounds naphthalene, monomethyl naphthalenes, acenaphthene, acenaphthylene, fluorene, phenanthrene and anthracene, primarily oil-derived compounds; HMW PAH includes the 4- and 5-ring PAH compounds fluoranthene, pyrene, benz[*a*]anthracene, chrysene, benzo[*a*]pyrene and dibenz[a,h]anthracene, primarily combustion-derived compounds. Although a wider suite of PAHs is determined routinely for both licensing and monitoring purposes, these can be considered as toxicity markers for the PAHs as a whole. The ERL and ERM concentrations applied are given in Table 3. The ERL and ERM values for LMW PAH are lower than those for HMW PAH as they have a higher acute toxicity. Using this approach no site in this study breached the ERL or ERM values (Table 3).

3.3. Sediment organohalogen contamination

PCB, PBDE and HBCD concentrations were determined in the sediments and reported on a dw basis. The ∑ICES 7 CBs (CB28, CB52, CB118, CB138, CB153, CB170, CB183), and the sum of all 25 measured CBs (∑CBs) were calculated and summarised in Table 4. Where individual congener concentrations were below the limit of detection (LOD), a value of half the LOD was inserted for calculation of summed concentrations (Supplementary data file 2).

Table 4
Organohalogen (PCB, BDE and HBCD) sediment contamination (ng g^{-1} dw). For ∑ calculations values below LOQ were replaced with 0.5 × LOQ.

Site name	∑ICES7 PCBs	∑25 PCBs	BDE209	∑11 BDEs	∑3HBCD
C1	0.35	1.25	8.879	0.22	0.69
C2	0.35	1.25	0.801	0.12	0.31
C3	0.41	1.31	0.977	0.11	0.19
C4	0.35	1.25	1.479	0.12	0.44
C5	0.35	1.25	1.254	0.14	0.47
C6	0.35	1.25	1.173	0.11	0.17
C7	0.35	1.25	<0.1	0.11	0.09
C8	0.35	1.25	<0.1	0.11	0.19
C9	0.35	1.25	1.073	0.12	0.40
C10	0.92	2	0.829	0.12	0.86
C11	0.35	1.25	0.82	0.13	0.25
C12	0.35	1.25	0.471	0.11	0.09
C13	0.35	1.366	0.177	0.15	0.13
C14	0.35	1.25	0.264	0.11	0.21
C15	0.35	1.25	<0.1	0.12	0.12
C16	0.35	1.25	0.546	0.11	0.14
C17	0.35	1.25	<0.1	0.11	0.09
C18	2.18	4.23	1.028	0.11	1.35
C19	0.35	1.25	1.01	0.11	0.20
C20	0.44	1.34	0.458	0.11	0.12
C21	0.35	1.25	<0.1	0.11	0.17
C22	0.35	1.25	0.13	0.11	0.12
C23	0.35	1.25	0.998	0.12	0.31
C24	41.91	78.4	5.7	0.14	0.56
C25	2.1	3.76	0.43	0.11	0.13
C26	7.3	14.07	7.92	0.35	1.27
C27	0.46	1.36	<0.1	0.11	0.09
C28	0.35	1.25	<0.1	0.11	0.22
C29	0.44	1.34	1.15	0.11	0.12

The spatial distribution of ∑ICES 7 CBs, ∑11 BDEs (BDEs 17, 28, 47, 66, 85, 99, 100, 138, 153, 154, 185), BDE209 and ∑3 HBCDs (αHBCD, βHBCD, γHBCD) in sediments is shown in Table 4 and Supplementary data file 4. ∑ICES 7 CB concentrations ranged from <0.7 to 42 ng g^{-1} dw, with concentrations at most stations <1 ng g^{-1} dw (Supplementary data file 2). The three highest concentrations of 42, 7.3 and 2.1 ng g^{-1} dw were at the closely grouped stations C24, C26 and C25, adjacent to the SIA a 'known' contaminated site in Kuwait (Beg et al., 2003; Gevao et al., 2006a,b). The only other result >1 ng g^{-1} dw was at C18, where the ∑ICES 7 CB concentration was 2.2 ng g^{-1} dw. Generally PCB concentrations were very low, with 20 out of 29 stations below the limits of quantification (LOQs).

Concentrations of PCB contaminants in the sediment were compared with various action limits, to investigate whether any adverse effects in benthic biota were likely to be expected as a consequence of their presence at levels detected with Kuwaiti sediments. When undertaking this analysis, PCB concentrations were normalised (to 2.5%) against the <2 mm TOC content (Table 1). For the purpose of this study assessment were made against the OSPAR Background Assessment Concentrations (BACs) and Environmental Assessment Concentrations (EACs) for the ∑ICES7 CBs in sediments (Table 5). Using OSPAR guideline concentrations below BACs would be considered to have high (excellent) environmental status. Concentrations significantly below EACs could be considered to have good environmental status and those above, bad environmental status (OSPAR, 2008). The station is deemed to have 'bad' environmental status if 'bad' status occurs for more than one ICES7 CB congener. Within the current data set 26 out of 29 stations were below BAC for all 7 congeners. The exceptions were C18 and C26, which had a few congeners between BAC and EAC, and C24 which was above EAC for CB101 and CB118. According to the OSPAR guidelines, most stations had 'good' environmental status for all ICES 7 CBs and 'good' status overall, except station C24, which had 'bad' environmental status for CB28 and CB118 and therefore 'bad' status overall.

The marine environment adjacent to the SIA has been subject to previous studies of PCB contamination. Similar to the data reported here Gevao et al. (2006a) documented elevated levels of PCBs (∑27PCBs 0.4 to 81.7 ng g^{-1} dw) at sites close to the SIA with the most abundant congeners being CB138, CB101, CB110, CB180, CB153, CB132, CB149, and CB118. Away from the SIA the depositional history of PCB sediment contamination has been studied in a number of cores collected from the western corner of Kuwait Bay (Sulaibikhat Bay). The sediment core data reflected deposition over 37 ± 5 year period, and demonstrated a peak in ∑PCB concentrations around 1991, after which values fall by 15 times to current surface concentrations of around 2 ng g^{-1} dw (Gevao et al., 2012). It was suggested by the authors that the 1991 peak was related to a sudden input of PCBs following the destruction of a number of electrical transformers during the final few months of the Gulf War (Gevao et al., 2012). Similar low levels of PCB contamination (∑PCB concentrations of 9.6–5.4 ng g^{-1} dw) have been reported by de Mora et al. (2010). The concentrations of PCBs in the majority of the 10 sites samples were not exceptional, and comparable to levels reported elsewhere in the region (Fowler, 2002; de Mora et al., 2004).

Table 5
OSPAR assessment criteria for ICES 7CBs in sediment (ng g^{-1} dw, normalised to 2.5% TOC).

Compound	BAC	EAC
CB28	0.22	1.7
CB52	0.12	2.7
CB101	0.14	3.0
CB118	0.17	0.6
CB138	0.15	7.9
CB153	0.19	40
CB180	0.10	12

∑11 PBDE concentrations ranged from <0.2 to 0.35 ng g^{-1} dw, with the highest concentration again seen at C26 (Table 4; Supplementary data file 4). The next highest concentrations at C13, C1 and C24 were all around 0.15 ng g^{-1} dw, and PBDE concentrations were also generally very low, with 17 out of 29 stations below LOQs. BDE209 concentrations ranged from <0.1 to 8.9 ng g^{-1} dw, with the highest concentration at C1 (see Table 4; Supplementary data file 4), and other high values of 7.9 and 5.7 ng g^{-1} dw at the SIA 'hotspot' sites C26 and C24. BDE209 was the 2nd most frequently detected organohalogen, with only 7 out of 29 stations below LOQs, and a median concentration of 0.8 ng g^{-1} dw. Previous work has also documented hotspots of PBDE contamination around Kuwait (Gevao et al., 2006b, 2014). Similar to the findings here Gevao et al. (2014) observed that BDE 209 dominated the mix, with similar levels detected in sites located across Kuwait Bay and the Gulf coast. Gevao et al. (2006b) also reported elevated levels close to the SIA. BDEs 153, 154 and 183 were most dominant forms of PBDEs detected with BDE 183 typically accounting for 60% of the congener mix. Values of PBDEs ranged from 0.08 to 3.8 ng g^{-1} dw, however it should be noted that this study did not report any information on BDE 209 (Gevao et al. 2006b).

∑3 HBCD concentrations ranged from <0.18 to 1.4 ng g^{-1} dw, with concentrations at most stations <0.5 (Table 4; Supplementary data file 2; Supplementary data file 3). The highest concentrations of 1.35, 1.27, 0.86 and 0.69 ng g^{-1} dw were at C18, C26, C10 and C1, respectively. HBCD was the most frequently detected organohalogen, with only 4 out of 29 stations below LOQs, however the median concentration of 0.19 ng g^{-1} dw was lower than that for BDE209.

4. Conclusions

Overall the data presented indicates that the majority of Kuwait's coastal and offshore marine environment is only exposed to low to moderate levels of contamination by the classes of contaminants studied. It further highlighted that, particularly for metals, the application of SQGs developed for other regions may not be suitable for use within the Gulf, due to the naturally high background concentration of metals present in the sediment. In relation to PAHs, PCBs, and PBDEs, hot spots of contamination were mainly associated with the SIA located to the south of the city. BDE209 and HBCD were also elevated at other locations around the coast and are new 'hotspots' for the region requiring further study.

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